Requirements for X-Ray Powder Diffraction System

Description

These requirements are for an X-Ray Powder Diffraction System (XRD) to be delivered by the contractor to the Naval Research Laboratory (NRL), Washington, DC.

The instrument must be capable of performing ambient and high temperature x-ray powder diffraction, in-plane and out-of-plane grazing incidence thin film diffraction, x-ray reflectivity and small angle x-ray scattering (SAXS) measurements.

The requirements for this instrument are not defined solely by scientific metrics, they are based on maintainability, usability and risk management to include simplified transition from one configuration to another. The instrument in question is not a specialty item to be used solely by one highly skilled operator. Instead, this instrument will serve multiple sections and possibly other divisions. As such, in a single week, multiple operators will use this instrument for multiple purposes. These users will have a variety of experience in XRD; some will have decades of familiarity with XRD, while others will have very limited experience. Therefore, these requirements, especially in regards to reconfiguring the instrument for different types of measurements, are designed to eliminate potential operator errors. Thus, whenever possible, the mounting and dismounting of the components must be minimized. Also auto-realignment is preferred over other more user dependent modes of reconfiguring the instrument.

Requirements:

1. Safety

1. Threshold Requirement: System must comply with all national radiation safety regulations. Objective Requirement: Additional consideration will be given for any documentation or other evidence of enhanced/improved radiation safety devices or protective capabilities.
2. An enclosure with a clear viewing window must be provided with safety interlocks with electromagnetic shutter mechanism which cut the x-ray radiation and freezes the run in the event the door is opened and continues the run without the loss of data when the door is closed. There should be a method to lock access to the x-ray goiniometer with a key. A lock for the enclosure will be sufficient for this requirement.

2. Goniometer

1. A theta-theta geometry which provides a simple solution for all the in-plane and out-of-plane diffraction measurements on all possible sample types including powders, thin films, bulk samples and liquids.

2. Accurate performance over the entire 2 Theta Range. Threshold Requirement is an accuracy of 0.05 degrees. Objective Requirement is 0.02 degrees of accuracy or better.
3. Collision or component conflict detection. Threshold requirement is either an alarm or computer notice when an operator attempts to perform a measurement that would either cause a collision between instrument components or when an improper measurement is attempted with a previous instrument configuration from a prior user. The system must determine the conflict and notify the user of the exact location of the conflict for easy correction. Instrument control software must be able to recognize all optics, display optical configuration, and automatically align optics.
4. Large angular range of motion up to 160 degrees two theta.
5. Accurate angle position detection across the entire 2-Theta Range.
6. Highly accurate 2-Theta movements. Threshold requirement is for 0.001 degrees 2-Theta. Objective Requirement is for 0.0001 degrees 2-Theta.
7. Fully automated alignment of the x-ray diffraction system. All the alignment results, such as x-ray source height, x-ray source angle, slit height, sample surface height and angle, alignment optics and detector, must be saved digitally for logging purposes. No manual alignments of the system or optics shall be needed at any time to optimize its performance.
8. In-plane diffraction. The goniometer must include hardware and software to perform in-plane scattering for examination of ultrathin films and full pole figure analysis with ease. Specifically, the goniometer must include capability to scan the detector in 2-axes – both in-plane (2-theta chi) and out-of-plane (2-theta) – to access a broad range of reciprocal space and obtain a whole pole figure. It must be possible to perform out-of-plane and in-plane measurements on both solid and liquid samples.

3. X-ray generator and Tube

1. Must include an x-ray generator of at least 3.0 kW.
2. Must include a Cu fine focus x-ray tube.
3. The desired voltage range is 20-60 kV with 2 kV or smaller incremental step and the current range is 2-60 mA with 1mA step.
4. Power stability must be provided. Both the voltage and current must be maintained with in ± 0.01 % against ±10 % fluctuations.
5. If the x-ray tube needs to be replaced, or another type of tube used, preference is given for a system which performs an auto-realignment of the tube.

4. Optics

1. The XRD system must include both Bragg-Brentano (BB) and Parallel Beam (PB) optics using x-ray mirror. Threshold requirement is for a system to be able to switch between these configurations with automated realignment. Objective requirement is to have both the BB and PB optics be mounted simultaneously

without having the user to mount or dismount parts while switching between the two.

2. Parallel beam optics. The incident parallel beam after the optics must have a divergence less than or equal to 0.04 degrees.
3. Diffracted beam monochromater for improving the quality of collected data. A curved graphite monochrometer for BB optics and a flat graphite monochrometer for PB optics must be included.
4. Slit controls. The requirement is that all slits (divergence, scatter and receiving slits) be computer controlled and programmable.
5. Changes to the optics. Threshold requirement is that the optics be simple to change for alternate applications or upgrades. The objective requirement is that this change be accomplished with automated realignment.
6. Small Angle X-ray Scattering (SAXS) optics. The x-ray diffraction unit must have SAXS optics for the determination of the microscale or nanoscale structure of particle systems in terms of such parameters as averaged particle sizes, shapes, distribution, and surface-to-volume ratio. The SAXS optical system must provide up to 100nm resolution and provide the pore size / particle size in 2–100 nm range. Sample stages must be provided for both transmission SAXS of bulk, powder, or solution samples and reflection (grazing incidence) SAXS to measure films on substrates. The system must provide a mechanism to reduce air-scatter and enhance the SAXS measurement performance.

5. Sample Stages

1. High precision sample stage. The system must include a high precision motorized sample stage for Z-axis sample height alignment. Threshold requirement for Z-axis movement to span over several mm with a minimum step size of 0.0005mm.
2. The sample stage must hold the sample horizontally for all the measurements except for the transmission mode measurements.
3. The system must include an automatic sample changer that allows the users to load multiple samples at a time and program to run the samples in a sequence without the operator's presence during the sample change.
4. The automatic sample changer must allow both reflection and transmission mode measurements. The objective requirement is that the automatic sample changer holds at least 6 samples.
5. Must include enough sample holders sufficient to fill the sample changer for both reflection and transmission measurements.
6. The sample stage and automatic sample changer must allow use of zero-background sample holders for x-ray measurements on milligram size powder samples.
7. Sample spinning mechanism must be provided during measurement.
8. A high temperature stage is required that:
    A. Can reach, at a minimum, a temperature of 1400 C.
    B. Must be a variable atmosphere system capable of working with air, inert gases or vacuum.

C. An oil-free vacuum pump must be provided.
D. A programmable temperature controller must be provided that allows multiple set points, ramp rates and hold times for the high temperature stage.
E. The control of the high temperature stage operations must be fully integrated in the x-ray control software.

6. Detector

Detector. A scintillation counter detector for use with all measurement applications and optical geometries must be included. The detector must be computer controlled with background less than 0.5 cps and detector linearity from 0.5 up to 0.5 million cps or better.

7. Software

1. All system operation such as data collection, automatic alignment, checking for conflicts among components, flagging the conflicted items and suggestions for action must be included in the software.
2. The application packages must provide turnkey operation and able to run in real time sequence and real time data display.
3. Data acquisition software must include preprogrammed application packages for specific measurement types with preset measuring conditions, optimum optical configuration settings, and guide user in set up, sample mounting, and measurements. Software must have capability to sense and recognize optics and display instrument configuration to user.
4. Full featured powder diffraction and general XRD data analysis software for analysis of diffraction patterns with a variety of display tools and functions, automatic data reduction of background removal, Kalpha2 stripping, peak top and second derivative peak finding, beam footprint and absorption corrections for variable slits and monochromators, Reference Intensity Ratio (RIR) quantitative analysis using imported phase information, percent crystallinity determination using whole pattern fitting method, crystallite size and lattice strain determination, instrumental broadening deconvolution using internal or external standards, cell refinement using internal or external standards, standardless cell refinement using theoretical systematic error correction, residual stress analysis, pattern indexing, indexing of an unknown phase in a mixture of a known phase(s), Search/Match qualitative phase identification compatible with ICDD PDF-2 or PDF-4 databases using a hybrid line and profile search algorithm with chemistry filters, and quantitative analysis, lattice parameter determination, and structure refinement using whole pattern fitting Rietveld method.
5. Pole figure data analysis and 2D/3D display of preferred orientation, topography or contour display with various color settings, overlay of multiple pole figures.
6. X-Ray Reflectivity (XRR) analysis and modeling software for film thickness, density, and surface roughness determination, reflectivity analysis and optimization of film thickness, density, and roughness using curve fitting, real

time display of reflectivity simulation, and Fast Fourier Transform thickness analysis.

7. Small Angle X-ray Scattering (SAXS) modeling and data analysis software to show, pore/particle size distribution function displayed in volume and number fractions. It must support both transmission and reflection (GI-SAXS) analysis modes. Software must model various shape functions to include spheres, rods, tubes, discs or plates and core/shell model and provide instrumental slit correction.
8. Up to 10 licenses of all the data analysis software must be included.
9. ICDD PDF-2 Database, Latest Release. At least one license must be included.
10. NIST/FIZ Inorganic Crystal Structure Database (ICSD), Latest Release, at least one license must be included.
11. The system must include a computer for system operation. Computer must allow remote access for on-board diagnostics and system control.

8. Water Cooling

A refrigerated, water-cooled chiller must be included with the capacity to cool an x-ray tube running at a full power. The chiller must be outfitted with the necessary hardware, such as alarms, flow meters, etc.

Pre-Installation Requirements

Along with submission of the proposal, the contractor shall submit a pre-installation guide that shall specific all site requirements including utility hook-ups (e.g. electrical, water, air, vacuum, etc.), required space (e.g. doorway passage, floor space, operating space, maintenance access space, etc.), procedures for receiving the equipment and placing it in its laboratory location.

Documentation

1. One (1) complete set of manuals for system operation/maintenance must be delivered with the system.

Delivery

1. Delivery of the complete system shall be no later than 120 days from date of award.

Installation

1. Contractor shall install equipment within 30 days of delivery.

Training

1. The contractor must send an XRD Applications Scientist to NRL, all expenses paid by contractor, to perform a minimum of 3 days of system training on system operation, sample preparation and mounting, use of all accessories, and software operation and data analysis.

Maintenance/Support

1. Vendor must provide standard Telephone Technical Support staffed by a dedicated team of technicians available during business hours.
2. Support must be available for service and applications.

Warranty

1. The system must include a 1-year warranty that covers all parts, labor, and travel expenses for on-site support of the equipment. The 1-year warranty will become effective after acceptance of the system.